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RADIATION EFFECTS ON POLYIMIDE FILM  
MATERIALS FOR HIGH TEMPERATURE APPLICATIONS  
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# **SIMULATION OF SPACE RADIATION EFFECTS ON POLYIMIDE FILM MATERIALS FOR HIGH TEMPERATURE APPLICATIONS**

**Final Report**

**by:**

**Lawrence B. Fogdall  
Sheridan S. Cannaday**

**Submitted to:**

**The Jet Propulsion Laboratory  
Pasadena, California**

**In Completion of Contract 954701**

**November 1977**

**Boeing Aerospace Company  
Seattle, Washington 98124**



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## ABSTRACT

Experiments have been conducted in simulation of vacuum and space radiation. The purpose of these experiments was to determine space environment effects on candidate materials for the solar sail film. Polymers, including metallized polyimides that might be suitable solar radiation receivers, were exposed to combined proton and solar electromagnetic radiation. Each test sample was weighted, to simulate the tension on the polymer when it is stretched into near-planar shape while receiving solar radiation. Exposure rates up to 16 times that expected in Earth orbit were employed, to simulate near-sun solar sailing conditions. Sample temperatures were controlled in one test; in another, they were allowed to reach equilibrium values under the influence of irradiation rate, solar absorptance and thermal emittance coefficients. Sample appearance, elongation, and shrinkage were monitored, noted, and documented in situ.

The relative performance of several polyimides was evaluated from the test results. Thermosetting polyimides tended to perform better than thermoplastics. That is, the former showed less degradation or visual change in appearance than the latter. In the first test (at controlled temperature), Upjohn 2080 and Ciba-Geigy B100 thermoplastic polyimides had more discoloration and greater length changes than Kapton (thermosetting). In the second experiment (free-standing samples), Ciba-Geigy thermoplastic polyimide samples underwent as much as 30% shrinkage, whereas shrinkage of Kapton samples was limited to 8% or less. This effort was performed under Contract 954701 between the Jet Propulsion Laboratory and the Boeing Aerospace Company.

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## Section 1

### INTRODUCTION AND SUMMARY

Two experiments have been conducted at Boeing's Radiation Effects Laboratory to simulate the radiation environment for a solar sail in interplanetary space, and to ascertain some of the effects of that environment on samples of materials considered for possible application on such a sail. A solar sail would likely be constructed of very thin polymer sheet (e.g., 2.0 to 2.5 micrometers thick) in order to minimize mass per unit area while maintaining adequate strength. The polymer would be metallized to a high reflectance, to maximize momentum transfer from solar radiation pressure to the sail and its payload (Reference 1). Propulsive force diminishes as the square of the distance from sun to spacecraft, so many solar sail applications are expected to be in the near-sun radiation environment for a significant portion of their mission life. The intensity of solar radiation could easily be 16 times that at Earth's distance from the sun, raising material temperatures in accordance with their absorbing and radiating properties. Section 4 of this report discusses the relationships of solar radiation, simulated radiation sources in the laboratory, and temperatures of test materials at equilibrium.

In Section 2 of this report there is discussed the first experiment Boeing performed, in which unmetallized polymers were placed in the vacuum chamber and irradiated with 1.3-keV protons and UV, visible, and IR radiation at an 11-sun rate. The purpose of this first set of tests was to ascertain the effects of such an environment on unprotected film, in the event there were areas in the sail where metallization might be missing because of problems occurring during fabrication, handling, stowage, deployment, etc. This represented a worst case analysis. Sixteen polyimide samples were "draped" over temperature-controlled cylindrical sections and weighted with ~1-gram masses, resulting in ~100-psi loading throughout the 300-hour irradiation period. Relatively small changes in sample length were documented using photographs of the weights' positions as a function of time. Simultaneously the irradiated sample faces were photographed to document changes in appearance. Portions of the samples' irradiated zones which were in contact with the 300°C copper cylinders were chemically altered and embrittled during exposure. More detailed presentation of test results is included in Section 2.



Section 3 of this report describes the second experiment or "second test stage" of the program, in which a more selective group of ten aluminized polyimide samples were irradiated with 1.3-keV protons and UV, visible, and IR radiation at a 16-sun rate. Test conditions differed from the first test set in that the ten samples were suspended so that their irradiated zones were "free-standing," i.e., not in contact with any temperature control mechanism, nor any portion of the vacuum chamber or other apparatus. Each sample acquired a steady-state temperature as determined by (1) its absorption and emission coefficients, (2) the proton and "UV" exposure rate, and (3) the rate of receiving secondary radiation from nearby surfaces. Again, each sample was weighted with a 1-gram mass, resulting in ~100-psi loading throughout the test, which lasted some 1100 hours. Appreciable reduction occurred in the lengths of 4 out of 5 samples made of Ciba Geigy polyimide. Lesser reduction in length occurred in all five DuPont Kapton polyimide samples. Detailed test findings are presented in Section 3.

Much high-quality photographic documentation of the condition of each sample took place before, during (in situ), and after each "test stage". Most of the transparency slides and enlarged photos for this documentation effort were delivered to JPL periodically during the program. Illustrations in this report summarize the test results and serve as examples of the types of in situ photo documentation undertaken. The printing of photos in this report is necessarily lower in quality than the original photographs, however.

## Section 2

### UNMETALLIZED FILMS EXPERIMENT

Both for this and for the other experiments conducted under this program, specialized combined radiation effects test chamber (CRETC) facilities at the Boeing Radiation Effects Laboratory (BREL) were modified to meet the test requirements. An "In Situ Mechanical Property Test Apparatus" was designed and fabricated to fit the CRETC II sample exposure chamber so that polymer test samples would face the incoming, combined beams from existing radiation sources placed around CRETC II. This sample apparatus is shown in Figure 1 before integration with the CRETC II vacuum chamber. Strips of flexible polymer film materials (Kapton and other polyimides described in Tables I and II) for subsequent mechanical property tests were

TABLE I. SOLAR SAIL FILM MATERIALS IRRADIATED  
IN BOEING CRETC II TESTS

FILM MATERIAL	SOURCE	JPL LOG	MEASURED <sup>③</sup> THICKNESS	TEST SAMPLE NO./TYPE
Plasma-etched commercial Kapton polyimide, etched side up <sup>①</sup>	NASA-Ames	141	0.165 mils	1
Solvent-cast Kapton-type polyimide, glass side up	NASA-Langley	66	0.06	2
Commercial Kapton, NR-150-B2G adhesive bond, pristine (unetched) side up	Dupont/JPL	118	----	3
Alkaline-etched commercial Kapton, 24-hr/600°F post cure, unetched side up	Dupont/JPL	82	0.07	4
Electro-cast polyimide, smooth side up <sup>①</sup>	TRW	135	0.055	5
Ciba-Geigy solvent-cast B100X polyimide, glass side up <sup>②</sup>	JPL/Ciba Geigy	140	0.300	6
Upjohn #2080 solvent cast polyimide, glass side up <sup>②</sup>	JPL/Upjohn	85	0.017	7
Commercial Kapton, no post cure, alkaline-etched side up	Dupont/JPL	106	0.147	8
① Thermosetting polyimide. ② Thermoplastic polyimide. ③ 1 mil=25.4 micrometers.				

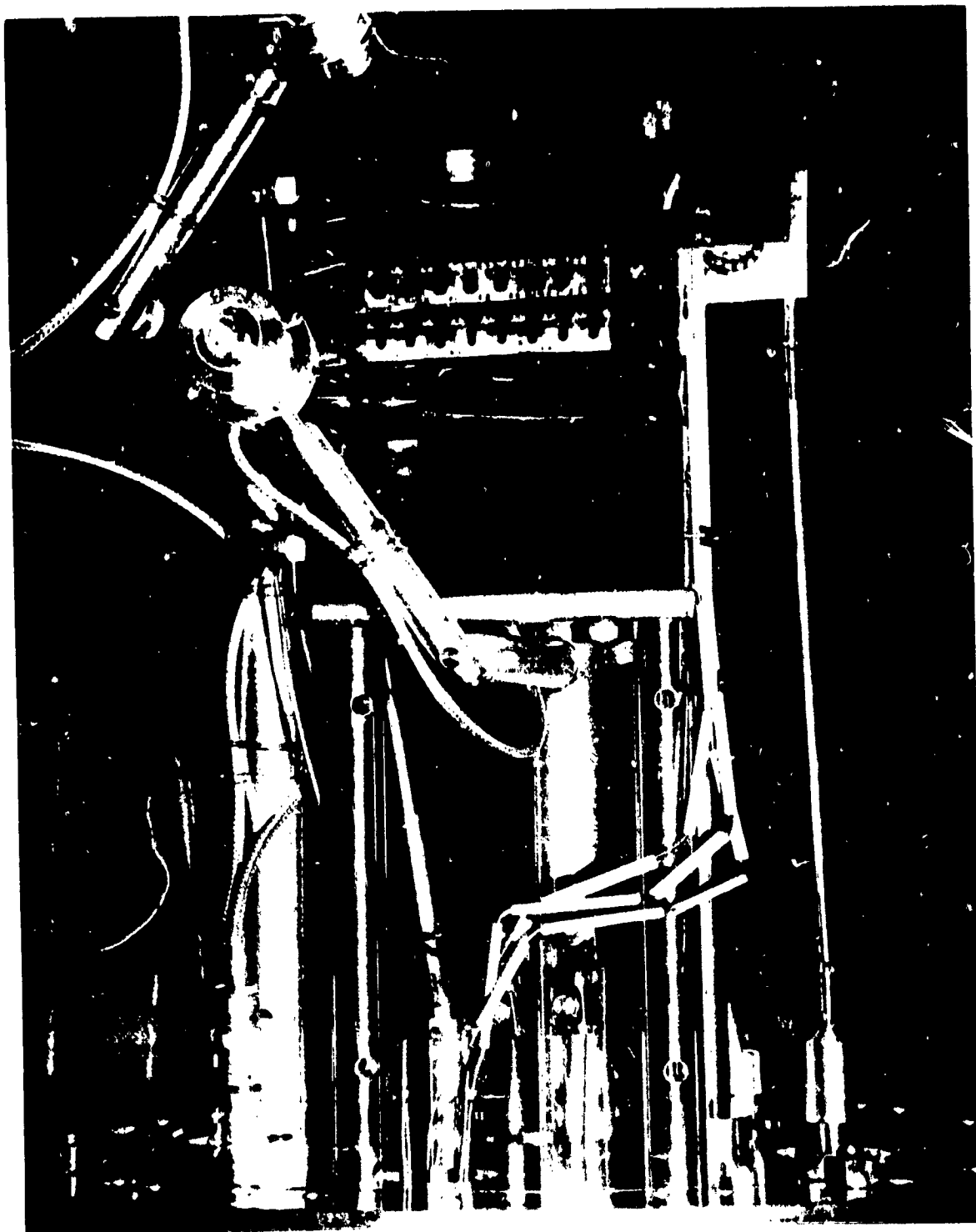


Figure 1. In Situ Mechanical Property Test Apparatus

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TABLE II. TEST LOADS IN SOLAR SAIL SAMPLES BEFORE RADIATION EXPOSURE

SAMPLE TYPE	MEASURED THICKNESS	SAMPLE WIDTH	CROSS-SECTION AREA	SAMPLE LOADING
1	0.165 mils ( $1.65 \times 10^{-4}$ inch)	0.219 inch	$3.61 \times 10^{-5} \text{ in}^2$	91.5 lbs/in <sup>2</sup>
2	0.06 mils	0.219	$1.31 \times 10^{-5}$	168 lbs/in <sup>2</sup>
3	(Joined sections - no single thickness)			
4	0.07	0.219	$1.53 \times 10^{-5}$	144 lbs/in <sup>2</sup>
5	0.055	0.219	$1.20 \times 10^{-5}$	147 lbs/in <sup>2</sup>
6	0.300	0.219	$6.56 \times 10^{-5}$	33.6 lbs/in <sup>2</sup>
7	0.17	0.219	$3.72 \times 10^{-5}$	59.2 lbs/in <sup>2</sup>
8	0.147	0.219	$3.22 \times 10^{-5}$	68.4 lbs/in <sup>2</sup>

placed in contact with the curved sectors of two cylinders shown in Figure 1. These cylindrical bodies were heated to and controlled at a temperature of +300°C throughout the test period. Each sample was made longer than the zone visible in Figure 1 so that ~1-gram masses could be suspended at the bottom of each sample to provide non-variable tensile loading of ~100 psi. Differences between projected sample thicknesses and actual measured values of film thickness account for sample loading less than and greater than 100 psi in Table II. The 16 masses with their pointers were visible from behind the test apparatus (see Figure 2) throughout the 300-hour exposure period of this "first test stage".

The CRETC II facility into which the sample apparatus was integrated for this program is shown in Figure 3.

Besides the 16 "strip" samples that were loaded by means of weights at their lower ends, 16 smaller ESCA test samples were placed in intermediate rows for exposure and chemical analysis later elsewhere. Figure 4 is a closeup photo showing all four rows, for a total of 32 test samples.

The basic plan involved shielding the top two rows of samples during the first 13 days of proton and "UV" exposure and then raising the shield for an additional day of exposure. The movable shield is shown "open" or "up" at the top of Figure 1. Thus, the top two rows of samples were exposed for one day and the

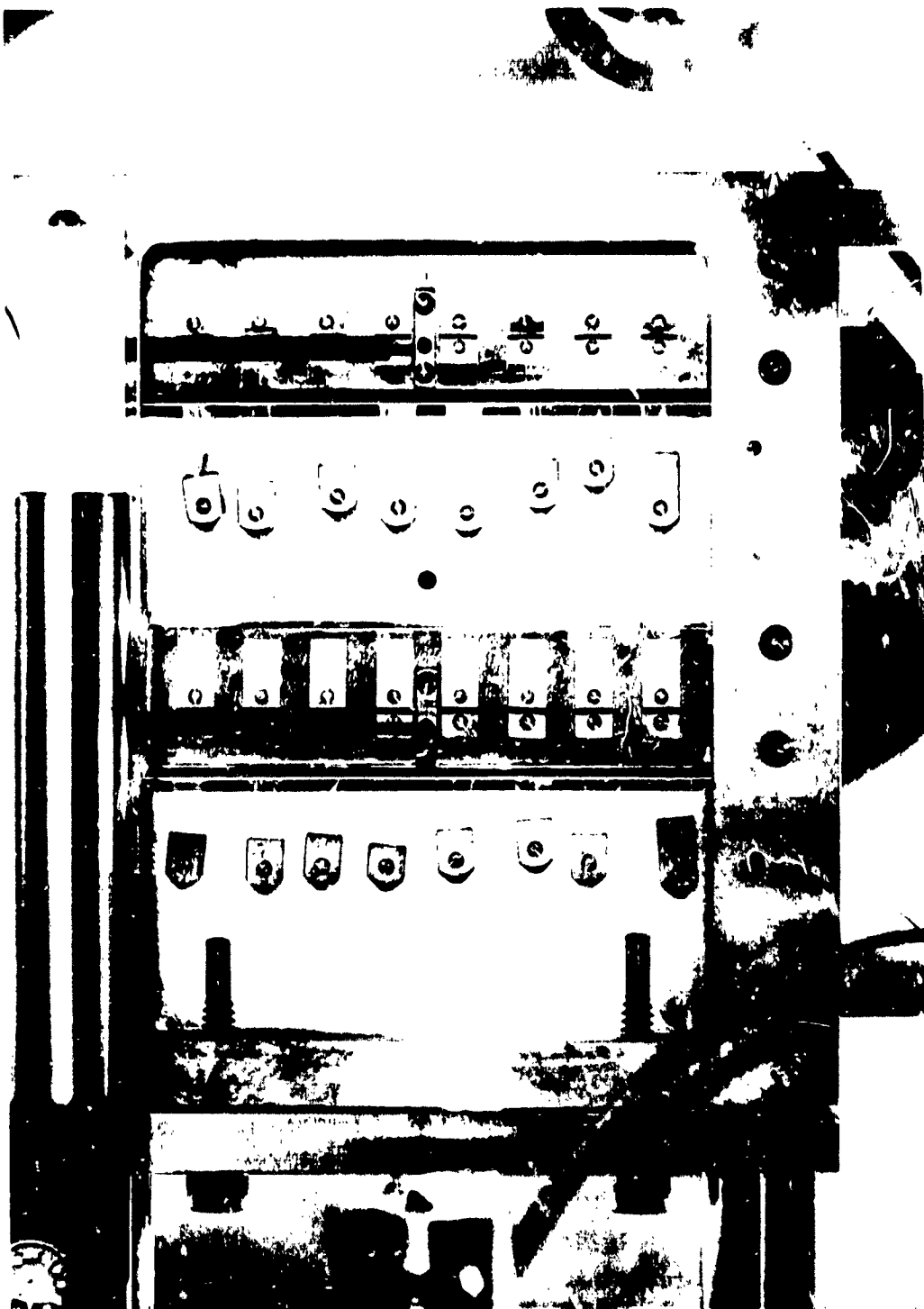


Figure 2. Weights Providing Tensile Loading of 16 Unmetallized Polyimide Films

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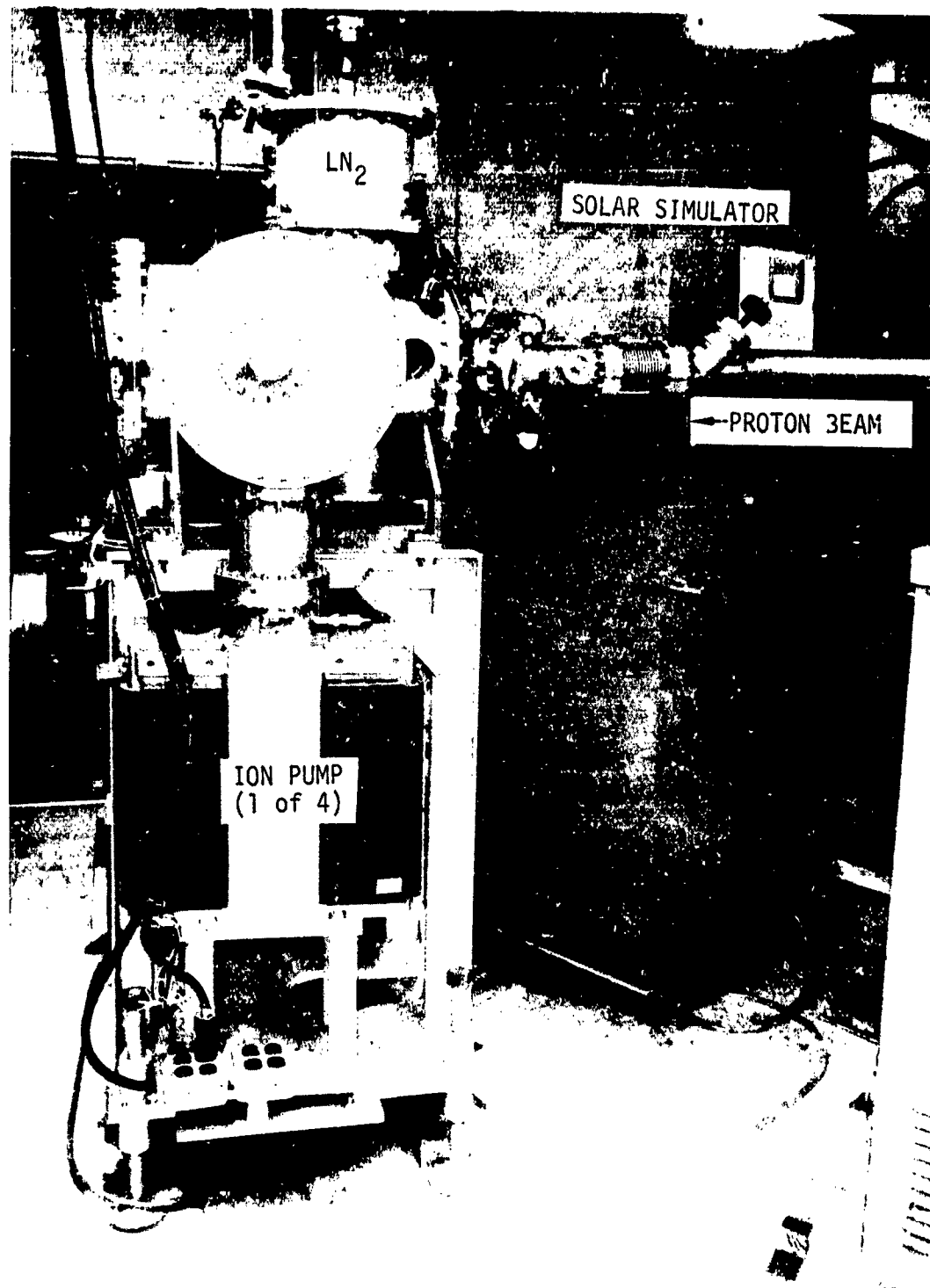


Figure 3. Boeing Combined Radiation Effects Test Chamber II and X-25 Solar Simulator

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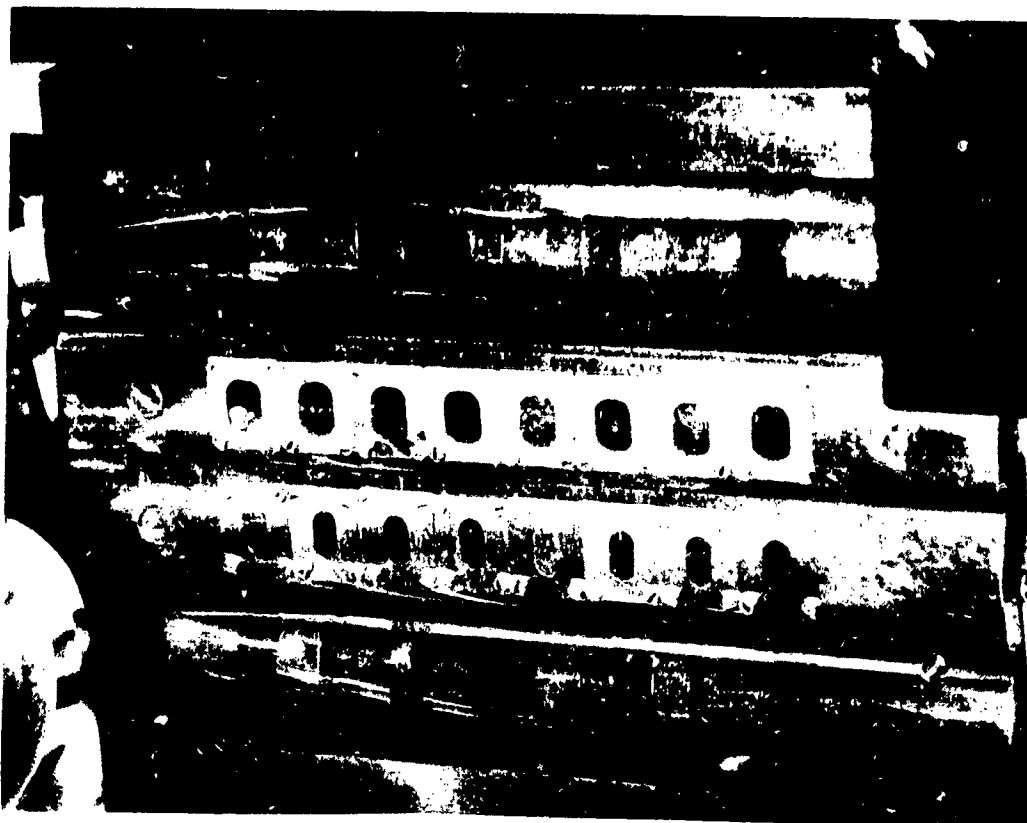


Figure 4. Array of Tensile and ESCA Test Samples Prepared From Unmetallized Polyimides.

bottom two rows received up to 14 days of irradiation. (Some of the sample area in the bottom two rows was partially out of the UV beam during the 14th day, as explained below further. The exact UV coverage of the bottom row of samples during the 14th day may be seen most precisely in photographs and slides previously delivered to JPL.) After this all samples were removed from the CRETC II facility for further analysis. A Spectrolab Spectrosun X-25 solar simulator was modified to produce 11-sun electromagnetic radiation, including the solar UV continuum ( $\sim 0.25$  to  $0.40$  micrometers, or  $5$  to  $3$  eV) from the simulator's xenon arc-discharge source. Figure 5 is a uniformity map of the simulator's output beam as measured during final calibration. The positions of the bottom two rows of samples during their exposure period of 13 days are shown as overlays on the simulator's output beam pattern in Figure 5. The "UV" beam was later shifted to expose the upper two rows

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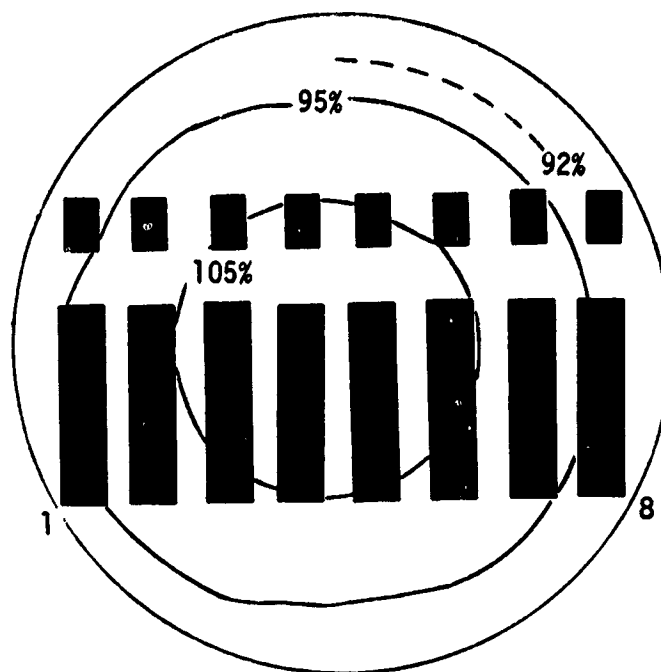


Figure 5. Uniformity of Ultraviolet Radiation (Actual Size of 11-Sun Beam)

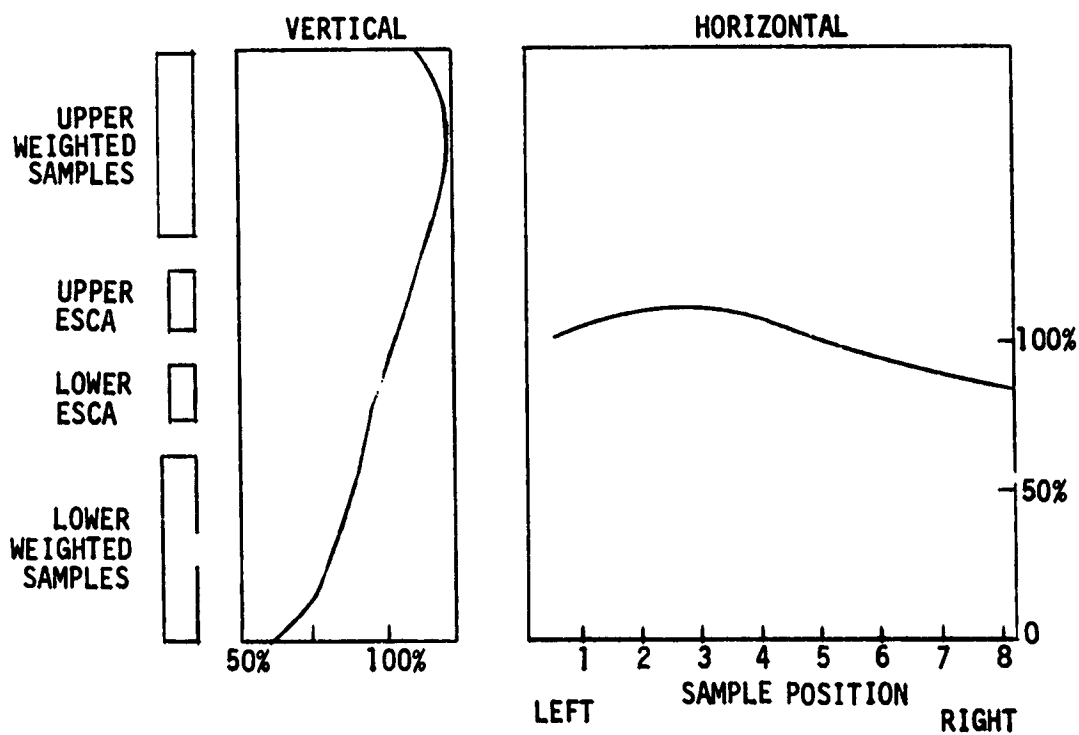


Figure 6. Proton Uniformity Along Near-Horizontal and Near-Vertical Arcs

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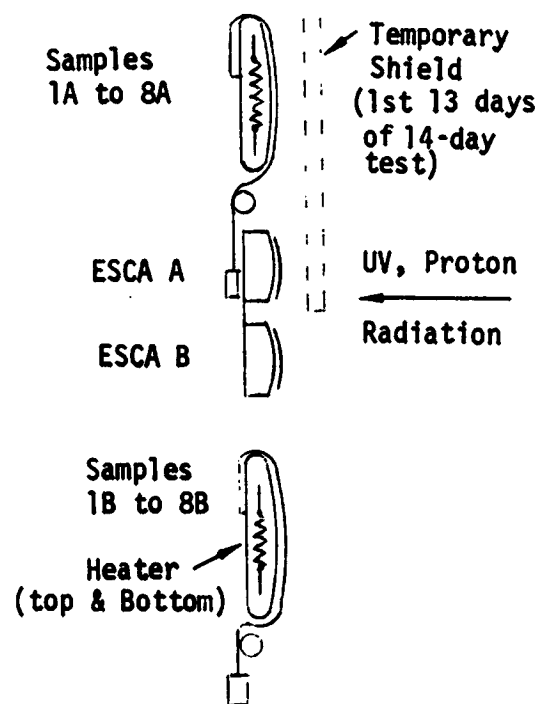
of samples, which if overlaid on Figure 5 would occupy an equal area.

The 1.3-keV proton beam from the CRETC II proton source was checked out relative to the sample array size. Figure 6 indicates the proton beam uniformity along the two Faraday cup tracks. The sweep of these tracks across the sample array is apparent from Figure 1. The "100%" proton radiation rate used during the "first test stage" was  $4 \times 10^9$  protons/cm<sup>2</sup>-sec. As seen in Figure 6, it is possible that the top two rows received somewhat more intense proton radiation.

Vacuum levels during the experiment were limited only by sample outgassing characteristics. Vacuum as measured by an ionization gauge ranged from  $10^{-8}$  to  $10^{-7}$  torr during peak outgassing periods.

Table I gives the type number (1 through 8) assigned by JPL to each prepared film. As stated before, the lower row of "strip" or weighted samples and the lower row of small samples — both called rows B — were exposed first while the upper two rows A were shielded.

A side view of the sample block would show, from top to bottom, an upper row of "strip" (weighted) samples; two rows of small, framed samples intended for later chemical analysis, behind which are the weights and reference lines for the upper row of strip samples; and the lower row of weighted samples. This sample arrangement is sketched at the right. Inside the uppermost and lowermost curved contact surfaces are electrical heaters, to provide the elevated test temperatures for the weighted samples. A thermocouple measures the temperatures of each row of weighted samples. A third thermocouple measures the temperature of the block on which the two rows of ESCA samples are mounted.



The lower two rows of samples (rows B) were first irradiated with UV and protons for 13 days, while the upper two rows of samples (A and ESCA A) were shielded from direct radiation exposure. However, the contact surfaces for all

strip samples (1A to 8A, and 1B to 8B) were at a temperature of 290°C to 300°C during this time. The ESCA samples' thermocouple indicated a temperature of approximately 290°C. During the first two hours of exposure, the weight attached to sample 7B (Upjohn 2080) rose approximately 70 mils. This was first observed by telescope sightings after 1.8 and 2.2 hours exposure. The weight for sample 6B (Ciba-Geigy B100) rose approximately 30 mils during this time period. The weight positions for all other samples in row B (the row being exposed) were unchanged. Visual and photographic observations of the front (exposed) side of samples 1B through 8B during this time interval indicate that material 7B darkened very quickly compared to all others, and that 6B's exposed-side visual appearance was unchanged until later in the test.

It is difficult to determine the effective sample length over which shrinkage (or stretching) would apply. By the end of the two-week test numerous locations in the exposed zones were observable at which polyimide material bonded to its heated copper substrate (the curved contact base). Thus, it is impossible to state with certainty the amounts of shrinkage (weight position change) as percents of sample length. The total shrinkage might or might not be uniform over the sample length from top to bottom. In some cases (see below, where exposed-side photographs are discussed) sample length change may be concentrated at the chemically altered "burn spots", "puckers", or "gathered" sites. The origin of these spots is postulated to be locations where there was lack of good contact to the heat-dissipating copper block.

It is possible to approximate the length of various samples suspended below their respective exposed zones. The group of reference lines below each row of weighted samples spans 16 times 40 mils, or 0.64 inch. Thus, the entire suspended length of the strip samples (below the exposure zone) is on the order of one inch. From this consideration we see that the shrinkage noted above is less than 10 percent of the sample's suspended length.

Further, smaller changes in sample length were observed as the test exposure period continued. Telescope sightings after 48 hours indicated: 7B shrank 10 mils more (total, 80 mils); 6B shortened 40 mils more (70 mils total); and other Row-B strip samples changed in length less than 10 mils. Furthermore, observation of the samples in Row A (heated but not exposed to UV or protons prior to opening their shield) revealed that sample 5A (TRW electro-cast) stretched 20 mils, as determined by its weight position, and the weight for sample 6A rose 30 mils.

Subsequent photographs and telescope sightings confirmed all these observations except the stretching of sample 5A, indicating (if it was a valid observation) it was transient whereas the others were permanent changes. Telescope sightings after approximately 90 hours and 130 hours exposure of Row-B samples indicated no further changes. The visual appearance of Row B samples as photographed after 13 days of irradiation by protons and "UV" is shown in Figure 7.

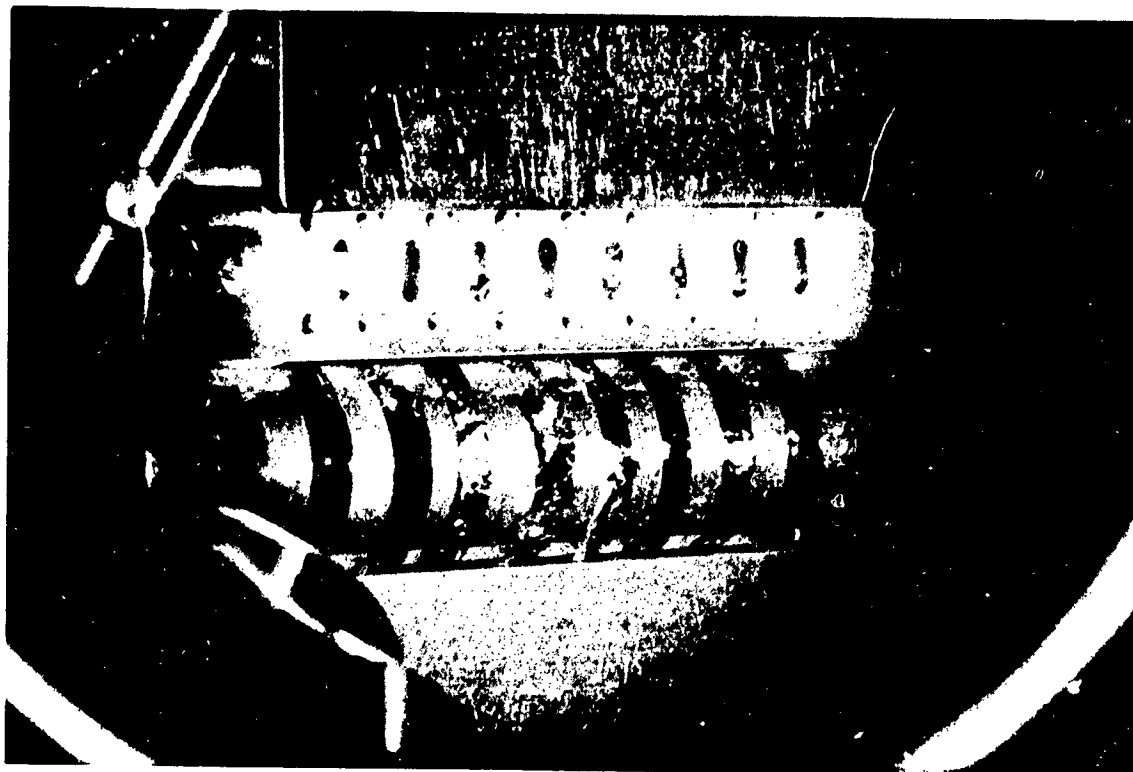


Figure 7. Effects of Combined UV/Proton Exposure of 16 Unmetallized Polyimide Film Samples for 13 Days (11-Sun Rate)

#### Exposure of Row-A Samples

The 16 polyimide samples in Rows A (8 tensile-test strips plus 8 ESCA) were irradiated by protons and UV at an 11-sun rate for 24 hours. Photographs of the irradiated film surfaces in contact with the cylindrical copper surfaces were taken frequently, to document the rate of thermal degradation. As with all other portions of the program, high quality color enlargements of the changes in these samples were forwarded to JPL. Figure 8 shows the appearance of these samples just after the beginning of exposure. Figure 9 is a photograph taken almost 24 hours later, just before the end of the 1-day irradiation.

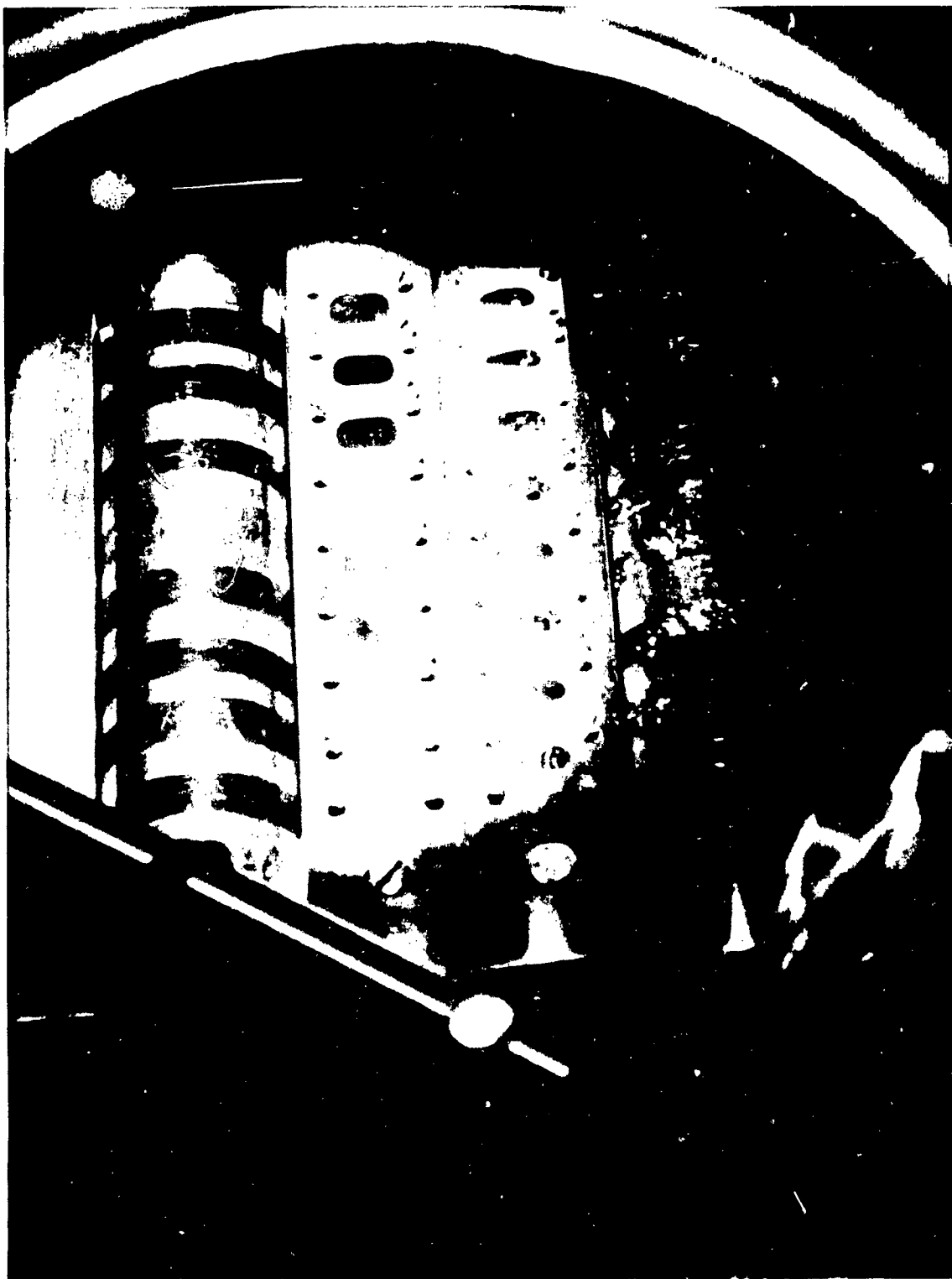


Figure 8. Unmetallized Polyimide Film Samples at Beginning of Irradiation.

UNMETALLIZED POLYIMIDE FILM  
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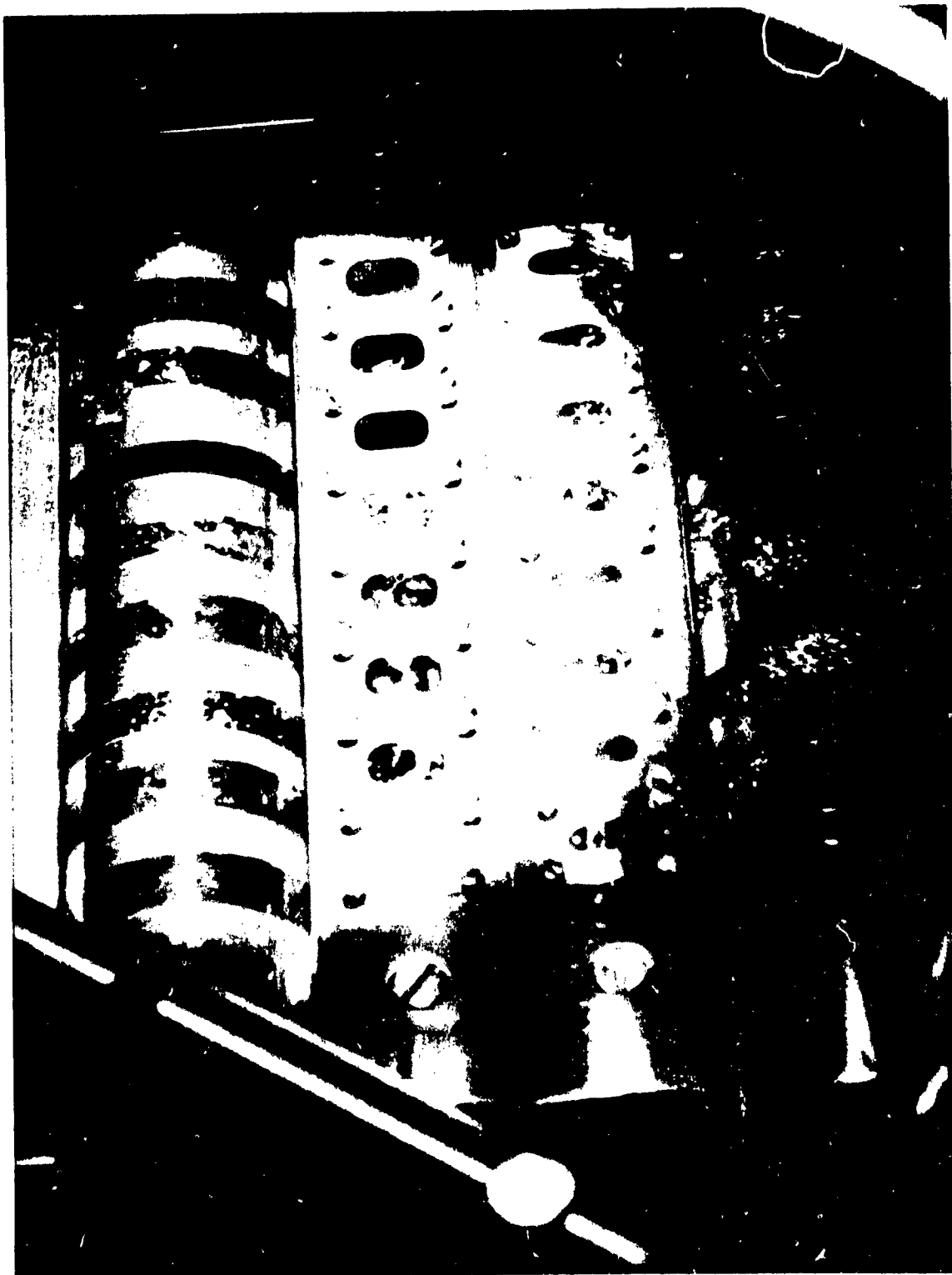


Figure 9. Condition of Unmetallized Polyimide Films After 1-Day Proton/UV Exposure.

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Regarding movement of weights below samples 1A through 8A, no significant position changes were observed 1.2 hours after beginning the combined UV/proton exposure. Within three hours after starting the irradiation, however, the weight below sample 3A (the bond joint) had risen approximately 40 mils and shifted slightly to the left as well.. This weight continued to rise throughout the 24-hour exposure, whereas the weights below the other seven samples remained relatively stable in their positions. Table III summarizes the position changes for the weight below this bonded Kapton film sample. Figure 10 shows the location of all 16 weights at the conclusion of the "first test stage."

TABLE III. WEIGHT POSITIONS OF TENSILE SAMPLE 3A

EXPOSURE TIME	RISE IN WEIGHT POSITION
0 to 1.2 hours	Baseline
3	Up 40 mils
5	Up 50
7	Up 60
8	Up 70
10 1/2	Up 80
14 1/2	Up 90
18 1/2	Up 100
23	Up 110

The darkening of various test samples during irradiation is thought to result from "carbonizing" of the materials during "thermal runaway." That is, as material temperature limits were exceeded they became more absorptive, leading to yet higher temperatures in the affected films. Thermal runaway was a major factor in enhancing the damage and rate of damage in affected samples.

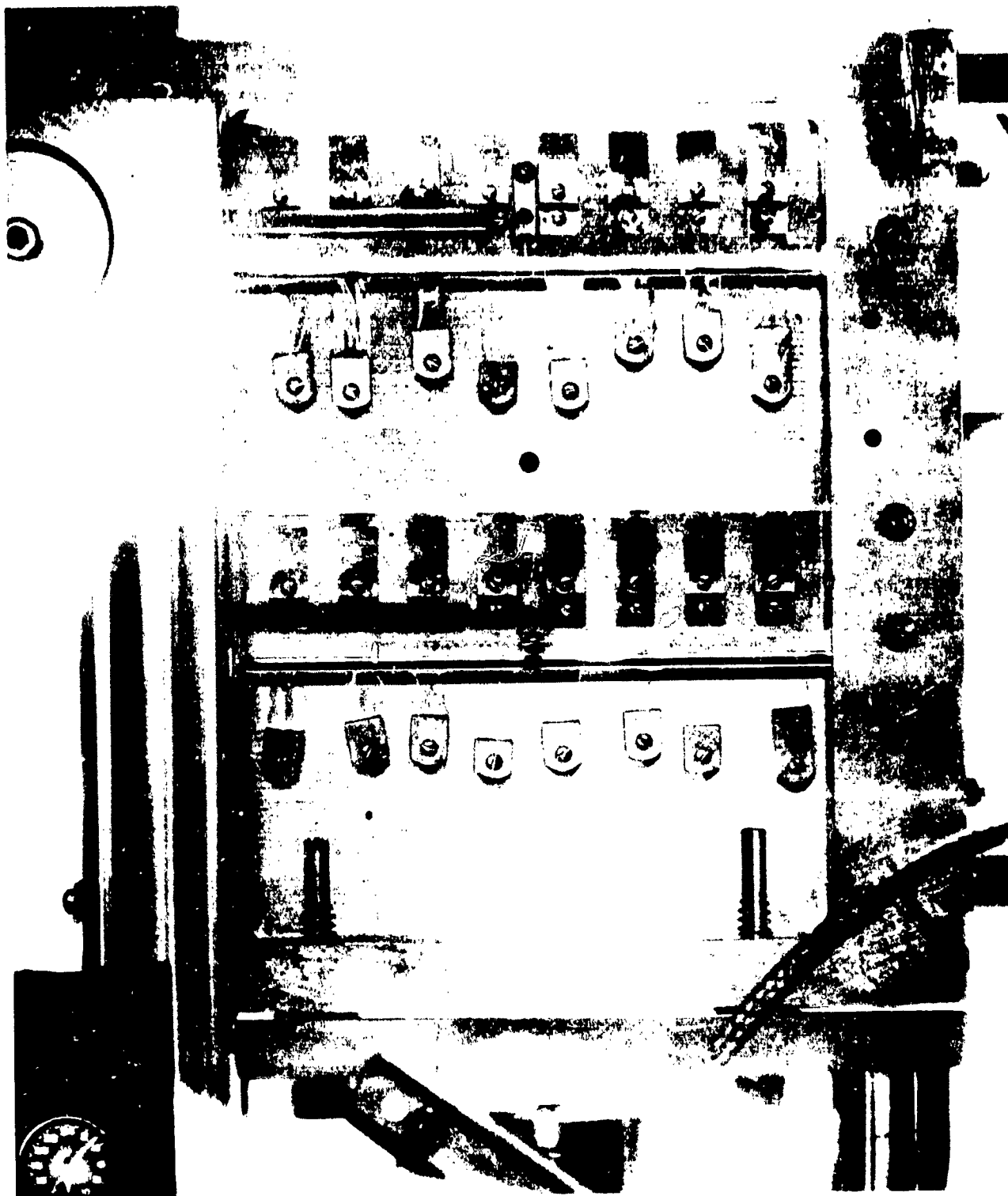


Figure 10. Positions of Weights Below 16 Tensile Samples After Unmetallized Films Experiment.

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### Section 3

#### METALLIZED FILMS EXPERIMENT

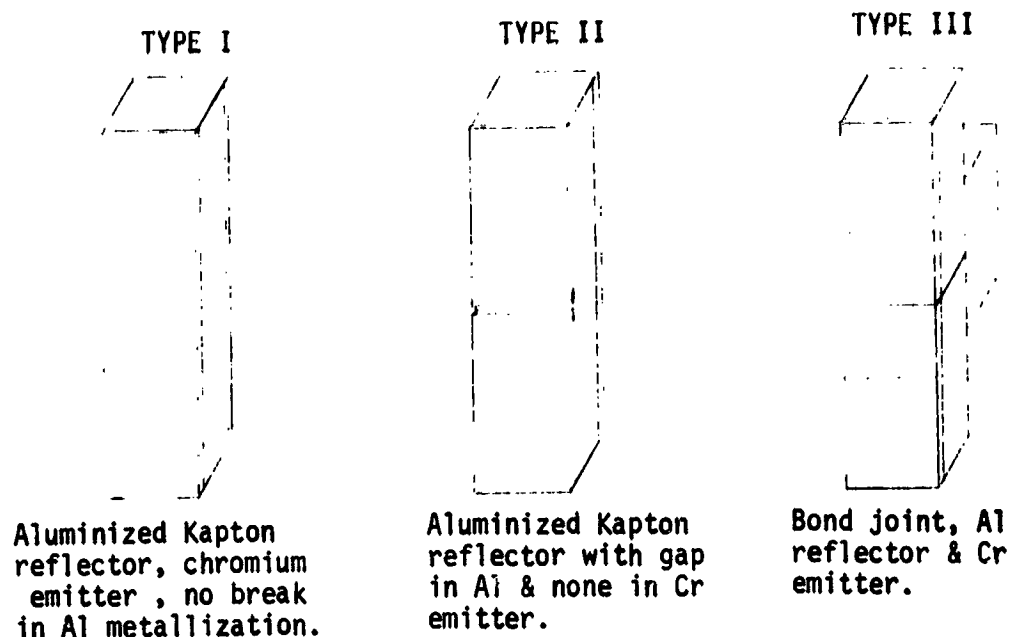
This experiment plus related effort to be discussed in Section 4 constituted the "second test stage" of this program. Boeing irradiated 10 metallized polyimide film samples with "UV" (i.e., simulated solar electromagnetic radiation including ultraviolet radiation) and with protons for nearly 1100 hours. The metallization on the samples consisted of vapor deposited coatings of  $\sim 125 \text{ \AA}$  of chromium (emitter) on one surface and  $\sim 1000 \text{ \AA}$  of aluminum (reflector) on the side facing the radiation. Unlike the previous experiment, these "strip" samples' entire lengths made no contact with other temperature-controlling surfaces. Rather, they exchanged radiative energy with a water-cooled plate behind the exposure zone, and acquired equilibrium temperatures dependent on their solar absorptance ( $\alpha_s$ ) and thermal emittance ( $\epsilon$ ) coefficients and the exposure rate. During all but the first 22 hours of this experiment, irradiation took place at a 16-total-sun rate, as determined by thermopile and calorimetric measurements. The proton exposure rate was also proportionally greater than in the earlier experiment, namely  $6 \times 10^9$  protons/cm<sup>2</sup>-sec. This value is approximately 16 "solar winds."

The metallized film materials are listed in Table IV. The numbers 9 through 18 were assigned by JPL, avoiding redundancy with the earlier experiment on unmetallized films (Section 2). The face of the Kapton and Ciba Geigy polymers to be irradiated had been aluminized by vapor deposition to a thickness of 100 nm except for specific gap areas on six of the specimens that allowed irradiation of the base polymer. The gaps were transverse to the lengths of the samples. Most samples had been cut to exposure size along (not transverse to) characteristic processing marks. The entire back surface of each sample had been vapor-deposited with chromium 10 nm thick to increase emittance. This coating system (of these thicknesses) is referred to as the "benchmark" coating.

Figure 11 is a closeup photograph of the 10 metallized films prior to irradiation. The photograph is taken from the side to be exposed to UV and protons. The gaps that were intentionally put in the aluminum overcoating by selective masks during vapor deposition are visible in some samples. Views of the reverse side of the sample apparatus as modified for this experiment (Figure 12) show the 1-gram mass below each sample and show horizontal grid markings that allowed sample length changes to be measured in situ during exposure.



TABLE IV. ALUMINIZED SAMPLES FOR SECOND TEST STAGE



POLYMER	DESCRIPTION	SPEC TYPE	JPL DESIGNATION	BOEING CHAMBER POSITION	SAMPLE THICKNESS
Direct manufactured Kapton Polyimide with Benchmark Coating <sup>1</sup>	No gap	I	340D	13	0.16 mil
	1-mil Gap	II	340A	17	
	2-mil Gap	II	340B	15	
	5-mil Gap	II	340C	9	
	Bond	III		11	
Direct manufactured (P.J. Sweitzer) Ciba-Geigy B100 Polyimide with Benchmark Coating <sup>1</sup>	No Gap	I	306D	14	0.12 mil
	1-mil Gap	II	306A	10	
	2-mil Gap	II	306B	12	
	5-mil Gap	II	306C	18	Transverse <sup>2</sup>
	Bond	III		16	

<sup>1</sup> Benchmark coating consisted of 125 Å of Cr on the emitting side of film and 1000 Å of Al on the reflecting (sun) side.

<sup>2</sup> This sample (#18) was cut for irradiation testing in a direction 90° from the direction of cut of the other Ciba-Geigy samples. The others were cut with the long direction parallel to the machine direction. This specimen was in the transverse direction. This sample was dimensionally much more stable than the other Ciba-Geigy samples. (See Figure 11).

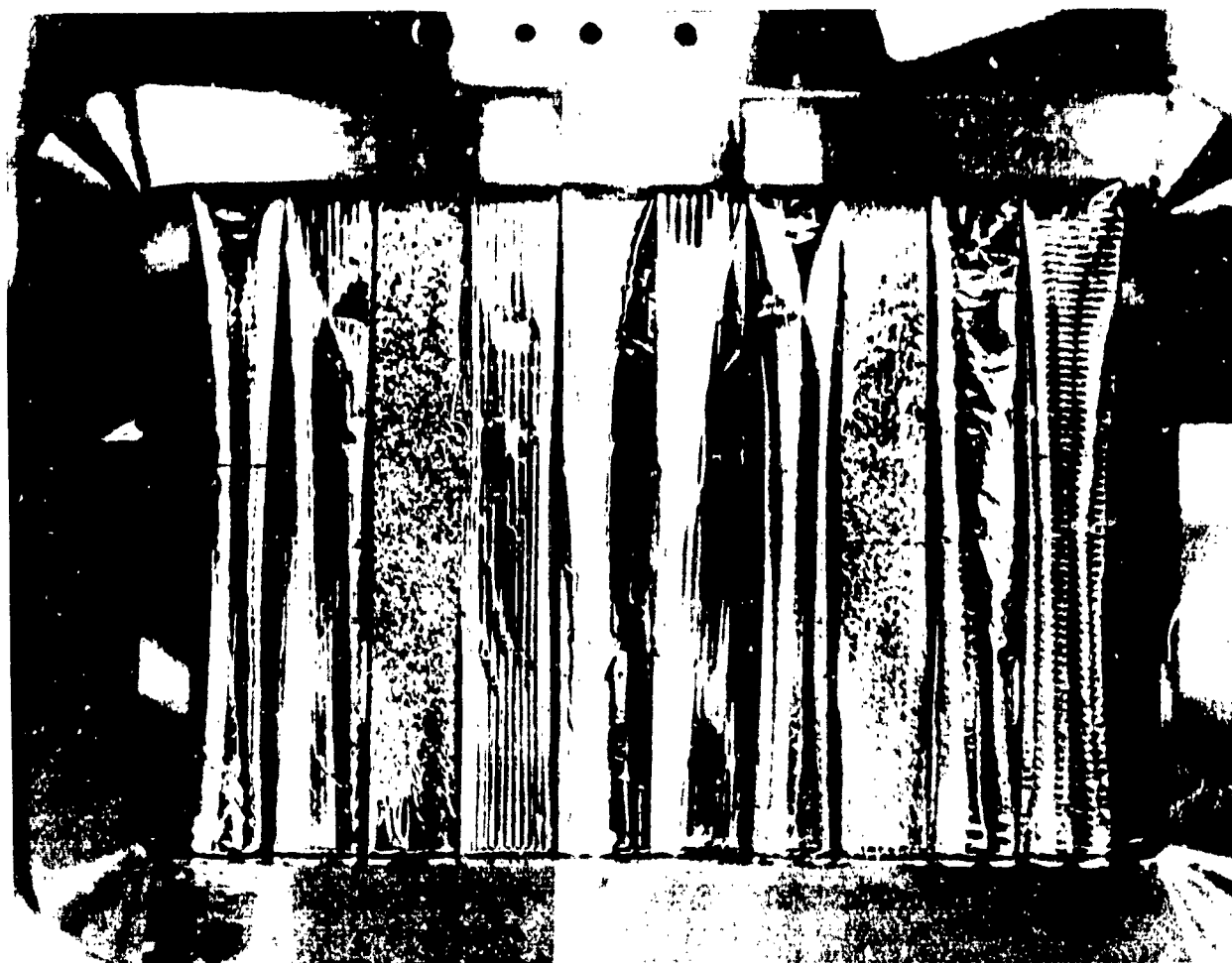


Figure 11. Metallized Polyimide Film Samples Before 16-Sun UV/Proton Irradiation.

Many of the details involved in setting "UV" exposure intensity and attempting the temperature calibration for the chamber are left for the discussion in Section 4 of this report. A principal objective was to expose the temperature-sensitive films to no more than 16 total suns. During preirradiation photography of the samples' initial conditions it was determined that the Ciba Geigy polymers that were "longitudinally cut" had already shrunk appreciably under illumination by the photo lighting source. Irradiation by the solar simulator continued the length shrinkage in these samples and to a lesser extent in the Kapton films. Data on sample length changes, as reduced from periodic photographs of sample weight positions in situ before, during, and after the 1100-hour exposure period, is presented for Kapton films in Figure 13 and for Ciba Geigy films in Figure 14. Since the current through the UV source lamp must periodically be increased to

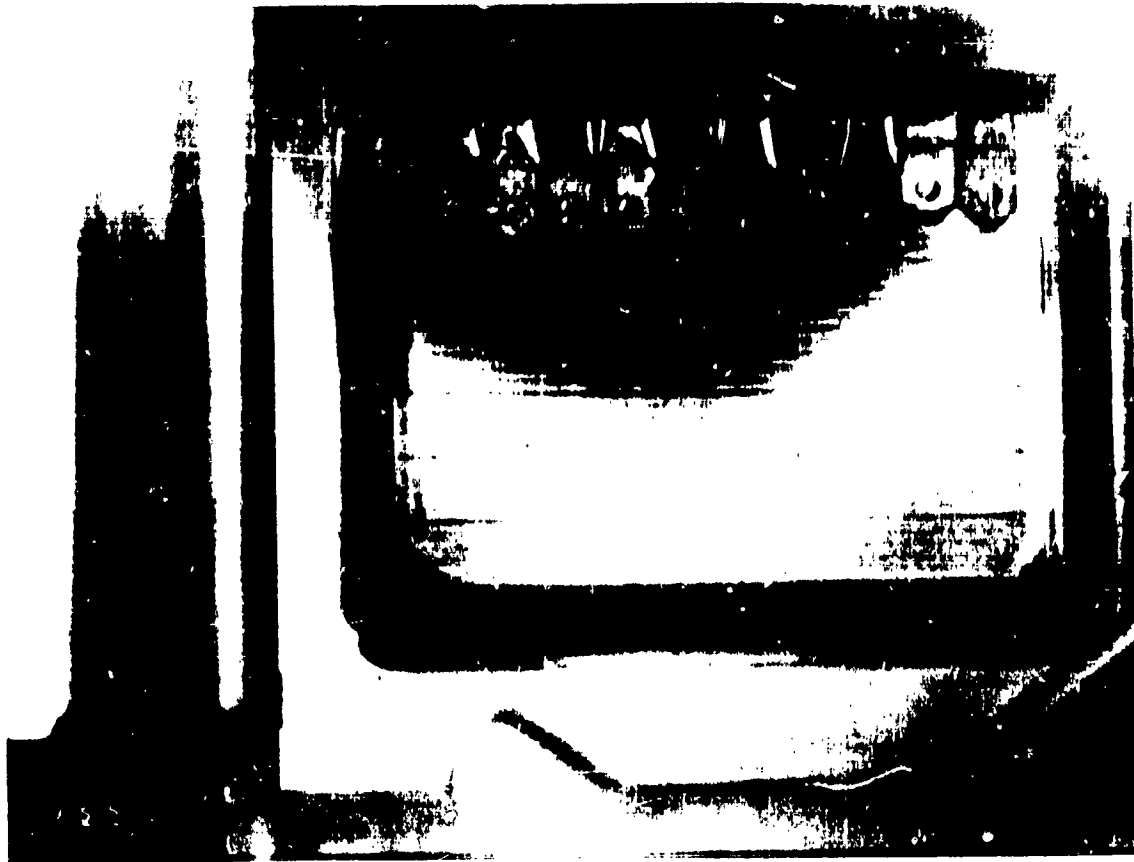


Figure 12. Weight Positions Below Suspended Metallized Polyimide Films Prior to Exposure

compensate for lamp aging, and since optical surfaces between the source lamp and the samples must periodically be cleaned, Figure 13 includes a charting of these adjustments to show the degree of correlation between small intensity adjustments and sample lengths. The main coincidence of sample length changes and solar simulator intensity increases is seen in Figure 13 to be after 22 hours of exposure when, as described in Section 4, the rationale for setting "UV" exposure intensity was modified by JPL. Figure 14 compares the stability of sample length among Kapton samples as a group, individual Ciba-Geigy samples cut longitudinally, and Ciba-Geigy sample No. 18 cut transversely.

The irradiated faces of all 10 samples were affected by proton/UV exposure, as shown in Figures 15, 16, and 17. The photograph in Figure 15 was taken at the beginning of the 1100-hour irradiation period, before "necking" or "dog-boning"

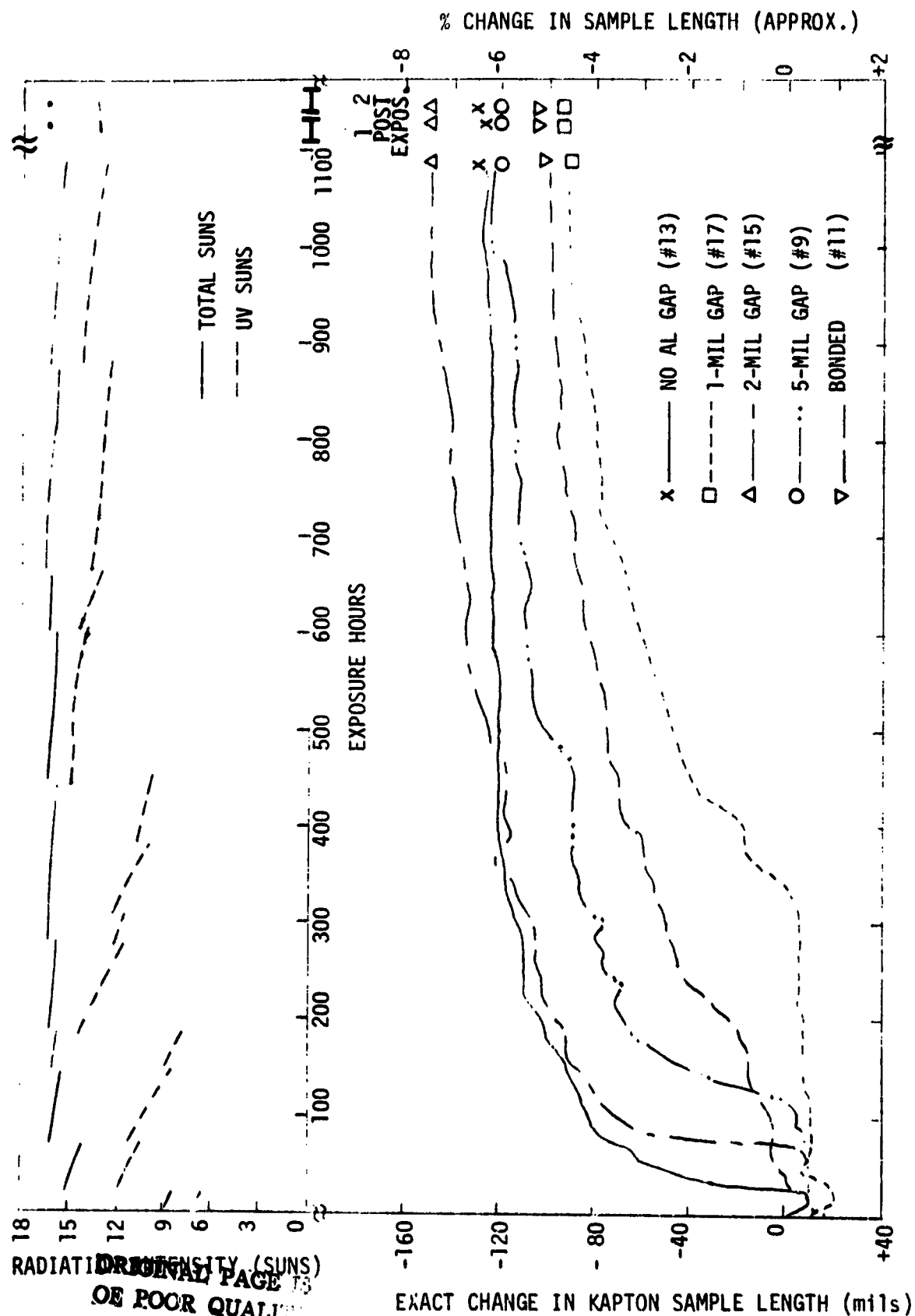


Figure 13. Changes in Radiation Intensity and Kapton Sample Lengths During 1100-Hour Test.

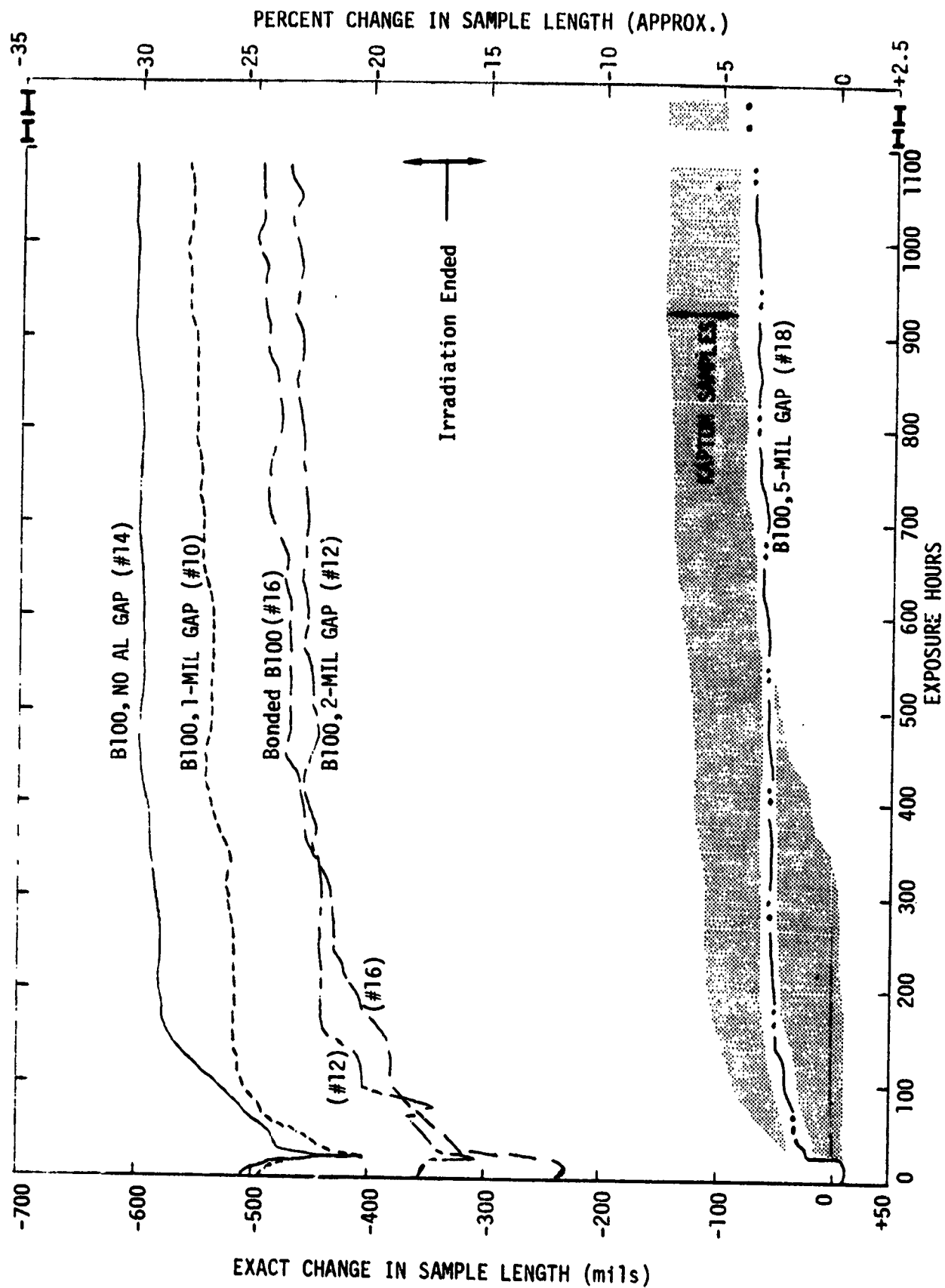


Figure 14. Changes in Metallized Kapton and B100 Film Lengths During 1100-Hour Test

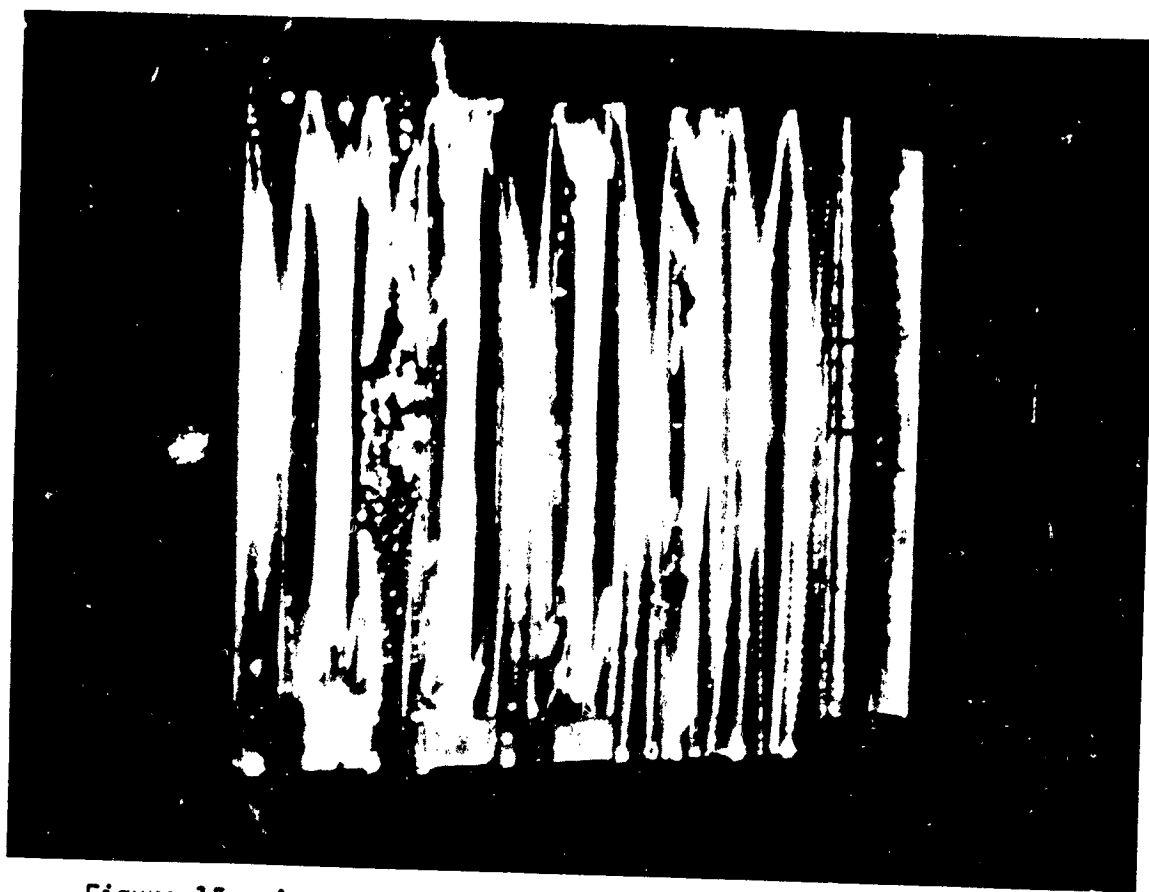


Figure 15. Appearance of Metallized Films Just After Start of 1100-Hour Test (9-Sun Intensity).

sample shaping began in the exposure zone. After the first 20 hours of exposure approximately 80% of the Ciba Geigy samples' irradiated lengths had narrowed, as (presumably) their transition temperature was exceeded. (Nevertheless, note these had undergone shrinkage, not elongation.) The transverse-cut Ciba Geigy sample (no. 18) appears in Figure 16 to be an exception to this statement about narrowing, although it did neck down soon after the exposure intensity was increased to 16 suns. The shape of the "transition line" (dashed lines in Figure 16) is not the shape of the solar simulator beam. (The shape of the solar simulator beam is indicated in Figure 18, and the uniformity of the proton beam is shown in Figure 19.) Toward the end of the experiment, extensive curling, warping, and twisting has affected both the Kapton and the Ciba Geigy materials, as shown in Figure 17.

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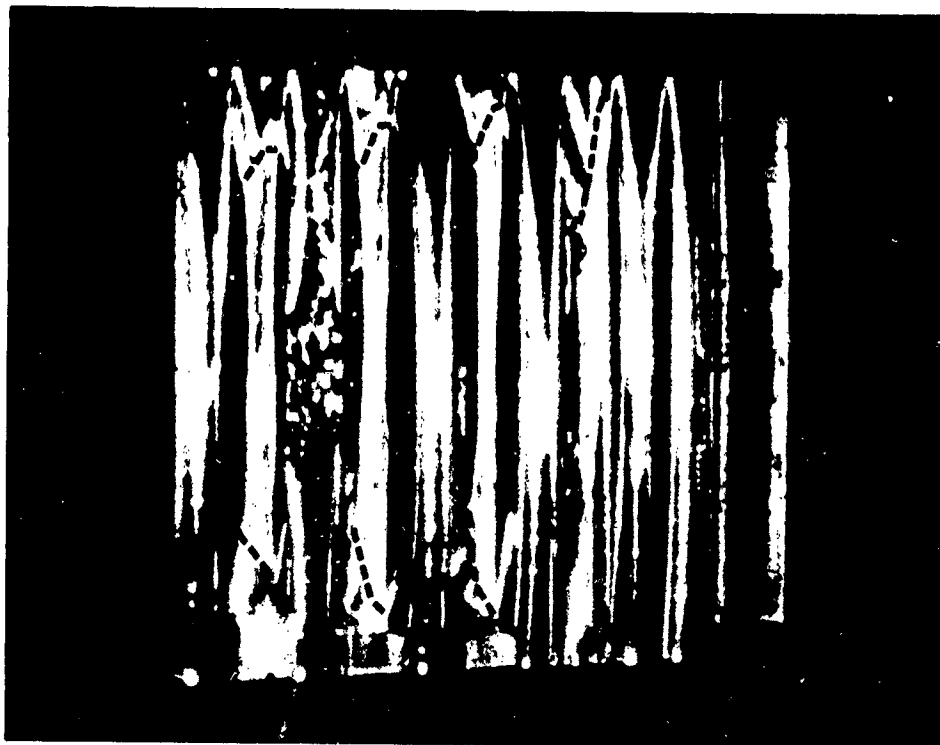


Figure 16. Metallized Films After 20 Hours Exposure.

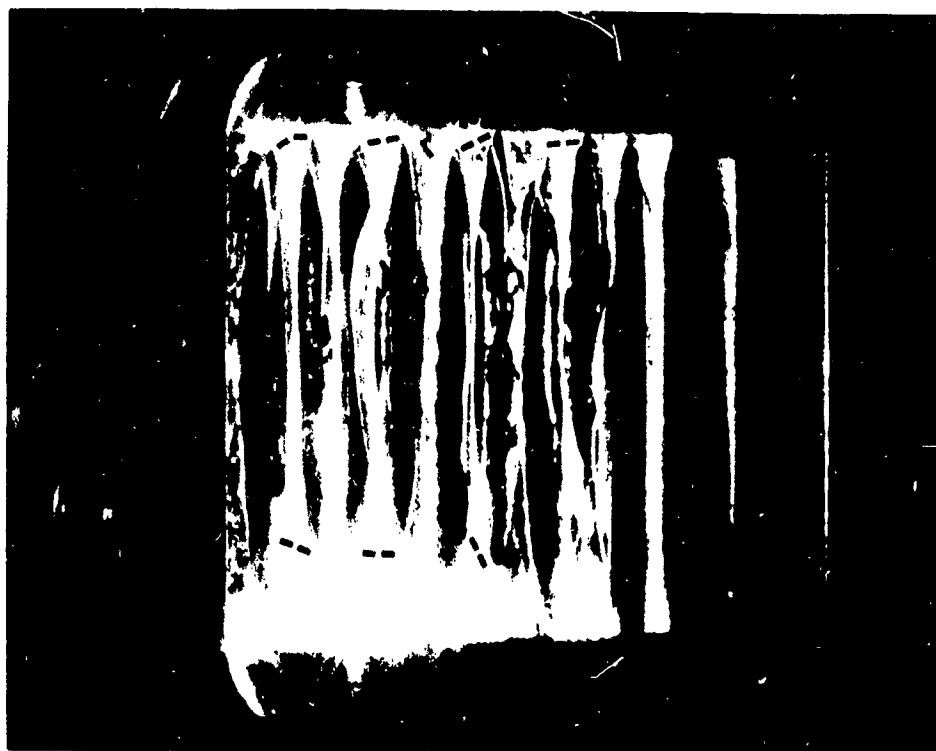


Figure 17. Metallized Films After 1000 Hours Exposure

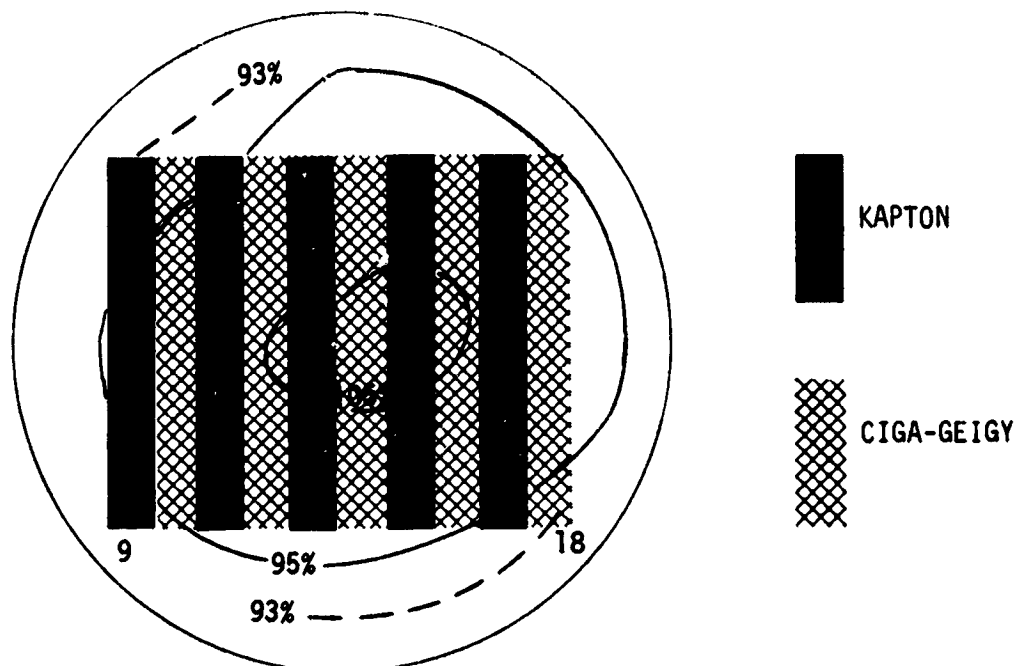


FIGURE 18. UNIFORMITY OF SOLAR SIMULATOR BEAM  
ACTUAL SIZE OF 16-SUN UV BEAM

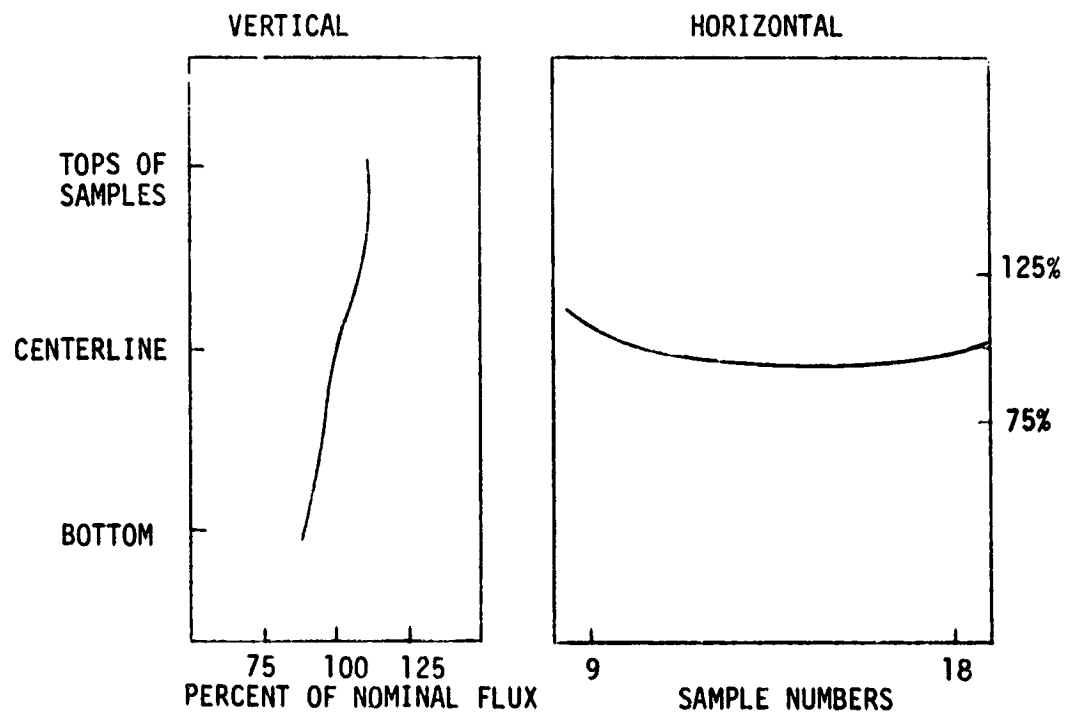


FIGURE 19. PROTON UNIFORMITY ALONG NEAR-HORIZONTAL AND NEAR-VERTICAL ARCS



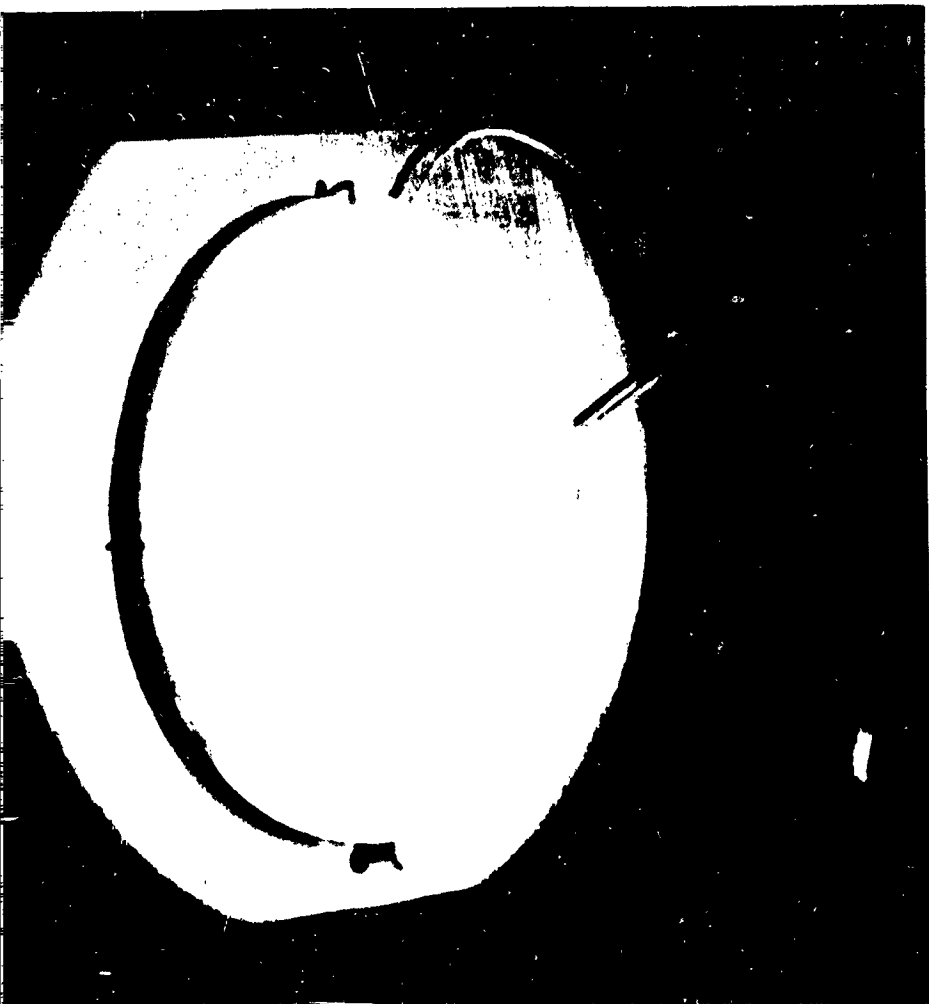
Combined narrowing plus shortening of a sample implies increase in material thickness (which was not measured) and/or loss of volatile molecules through outgassing in the vacuum test chamber. Vacuum gauge indications confirm the latter.

Thermal conditioning of the Ciba Geigy test samples before delivery to Boeing for irradiation may have been a factor in their greater instability. The Ciba Geigy test samples were "soaked" at 250°C in vacuum for 5 minutes. JPL believes that a longer conditioning period might have improved the dimensional stability of the Ciba Geigy material tested.

## Section 4

### IRRADIATION CALIBRATION EXPERIMENT

was designed by JPL and Boeing to compare solar illumination in the laboratory with the intensity of radiation anticipated for a Mars mission. A series of experiments was eventually performed, by which the results of doing UV dosimetry in the laboratory were checked against a standard that would have solar absorptance ( $\alpha_s$ ) and thermal emittance ( $\epsilon$ ) similar to those of the candidate solar sail film materials. The standard was mounted to Boeing and installed inside the CRETC II sample chamber, in front of the test film sample plane (Figure 20).



Aluminized Kapton Calorimeter Exposed to 16-Sun Solar Simulator Beam.

A solar sail film in a plane, receiving radiation on its front side and emitting radiation on both sides, would equilibrate at a temperature  $T$  such that

$$T^4 = \frac{\alpha_s (SC) (SR)}{\sigma(\epsilon_f + \epsilon_b)} ,$$

where  $SC$  is the solar constant,  $SR$  is the sun rate, and  $\sigma$  is the Stefan-Boltzmann constant. It can be shown that solar sailing under near-sun conditions (namely,  $SR = 16$  times the solar intensity at Earth's orbit) results in an equilibrium temperature of approximately  $+250^\circ\text{C}$  for aluminized Kapton that has an emittance-enhancing coating on the back side. On this basis a calorimeter closely simulating a solar sail film in  $\alpha$  and  $\epsilon$  characteristics could be used in a vacuum chamber to set the intensity of radiation from a solar simulator, provided secondary (bounce) radiation in the chamber were comparable to view factors (spacecraft geometry) in space.

The calorimeter discussed above is a circular disk with a diameter of two inches and thickness of 0.1 inch. Hence it approximates a solar sail film in a plane. It was fabricated by TRW with 2 chromel-alumel thermocouples on the unirradiated side, one at the center and the second 0.707 inch from the center — the radius which divides the calorimeter's total area into halves. Assuming edge losses of heat are small around the perimeter of the calorimeter, its thermocouple(s) will indicate the same equilibrium temperature as stated above for the case of irradiation and partial absorption on its front side and thermal emission from both sides (front and unirradiated back).

The irradiation source must be discussed from the standpoint of its spectral content, since absorption on the front face of a solar sail or the calorimeter varies with the wavelength. It is presently impractical to obtain total irradiation levels like 16 suns (to simulate near-sun solar sailing trajectories) along with "close filtering" spectral matches using a simulator mobile enough to be combined with an ultra-high-vacuum chamber and charged particle accelerator. (That is, the nearly immobile X-75 and X-200 style solar simulators that have enough energy to trade for close spectral filtering are not currently available, and the more mobile, X-25-type power limitations must be settled for.) As a result, the relatively large emission from a xenon arc in the near infrared (0.8 to 1.2 micrometers) is utilized along with the desired continuum across the visible and near-ultraviolet wavelength regions. Table V shows the relative spectral energy applicable to the start of

exposure of metallized film samples (the second principal experiment for this program, described in Section 3 of this report).

TABLE V. X-25 SOLAR SIMULATOR RELATIVE OUTPUT

BANDWIDTH (micrometers)		SCALED DATA	DATA MATCHED TO 1 SOLAR CONST. (watts/m <sup>2</sup> )	ENGINEERING STANDARD (watts/m <sup>2</sup> )	DEVIATION
0.25	0.35	4.7	27.3	58.5	-53.3
0.35	0.40	06.7	38.9	56.9	-31.6
0.40	0.45	8.5	51.3	86.8	-40.9
0.45	0.50	10.7	64.6	100.9	-36.0
0.50	0.60	22.8	137.7	177.0	-22.2
0.60	0.70	22.4	135.3	151.5	-10.7
0.70	0.80	19.5	117.8	123.6	- 4.7
0.80	0.90	33.0	199.3	99.3	+100.7
0.90	1.00	32.2	194.5	82.6	+135.5
1.00	1.20	22.2	134.1	120.7	+11.1
1.20	1.50	18.1	109.3	111.8	- 2.2
1.50	1.80	10.5	63.4	66.9	- 5.2
1.80	2.20	8.6	51.9	43.8	+18.5
2.20	2.50	4.1	24.8	19.9	+24.6

The three bands with the longest wavelengths partially represent emission from the incandescent electrodes of the xenon arc source lamp. This we eliminate by insertion of a water window between the solar simulator and the sample exposure chamber, since pure water absorbs wavelengths longer than ~1.4 micrometers and transmits shorter wavelengths down to approximately 0.2 micrometers. "Scaled data" represents energy arriving at the detector on a modified Beckman DK-1/spectro-radiometer measuring system. This data is then matched to the spectral shape of the solar constant in space (air mass zero) in accordance with NASA/IES engineering standards. The deviations of spectral power obtainable, compared with the ideal represented by latest measurements of actual solar output, are included in Table V.

Between the first and second test stages for this program, Boeing modified the in situ mechanical property test apparatus to provide for insertion of the disk calorimeter in front of the sample plane, and removal at any time using an in situ remote manipulator. As the metallized film experiment was about to begin, the calorimeter disk was moved into the solar simulator's "UV" beam. The group of 10

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test samples intervened between the calorimeter's emitting back surface and the water-cooled sample block, modifying the effective emittance of the calorimeter an unknown amount. It was determined that the solar simulator needed to be set at an output level considerably lower than that indicated as 16 suns by a pyrheliometer, if a calorimeter equilibrium temperature of +250°C or below were to be maintained. A pyrheliometer commonly used for this type of dosimetry then indicated that approximately nine total suns and seven UV suns were incident on the sample (or calorimeter) plane under these conditions. (See Figure 13).

After 22 hours of exposure the decision was made to return to the usual pyrheliometer dosimetry as a basis for setting solar simulator output intensity. This decision acknowledged that the presence of an array of test samples between the calorimeter and the water-cooled sample block effectively altered the  $\epsilon$  properties of the calorimeter so that its indicated temperature should not be used as a basis for setting the solar simulator's output level. The solar simulator output was accordingly increased to an intensity of 16 total suns as determined by pyrheliometer dosimetry readings. This change is shown in the top portion of Figure 13. The calorimeter was not reintroduced into the "UV" beam at this time, because it was known that the solar simulator intensity was now great enough to degrade the calorimeter materials thermally. That is, previous short-time insertions of the calorimeter into such an intense beam had shown by the slope of the response curve on a strip chart recorder that an equilibrium temperature substantially above +330°C would be reached.

At the end of the metallized films (Test Set No. 2) experiment the solar simulator output level, which was still being maintained at 16 total suns using pyrheliometer dosimetry, was again compared with indicated calorimeter temperature. As Figure 13 shows, the output level of the solar simulator had been adjusted and the optics cleaned from time to time throughout the 1100-hour exposure period of the "second test stage." The final output dial settings, not easily relatable to the output settings at 22 hours, understandably also resulted in calorimeter temperature indications too high to be sustained safely. The array of samples at test end was quite twisted and irregular in shape, as shown in Figure 17. Portions of the back surfaces of some test samples can be seen facing the radiation sources. In such areas greater absorption occurred, due to the existence of the emissive coating. This led to higher temperatures than planned for the specimens. The twisting modified the calorimeter's effective emittance to an unknown and different degree than at the start of the experiment. It was determined that the only precise data that could be

obtained would be a comparison of the calorimeter's temperatures for cases of samples present and samples absent (for a certain reduced simulator output level). Subsequently an equilibrium temperature of  $+334^{\circ}\text{C}$  was obtained from the calorimeter with samples still present and a certain solar simulator dial setting. After the ten test samples had been removed a calorimeter thermocouple temperature of  $+330^{\circ}\text{C}$  was obtained with the same dial setting. All temperature readings noted here were obtained using the thermocouple at the center of the calorimeter's unirradiated side. Readings from the thermocouple closer to the unirradiated side's edge were consistently  $3^{\circ}\text{C}$  less.

With the ten test samples removed, an outgassed film was clearly observable on the water-cooled sample block just behind the test sample plane. The patterns of outgassed material were typical of thin film interference coatings. As with all other phases of the program, high quality color photographs were taken to document the condition. Color enlargements were subsequently forwarded to JPL. Figure 21

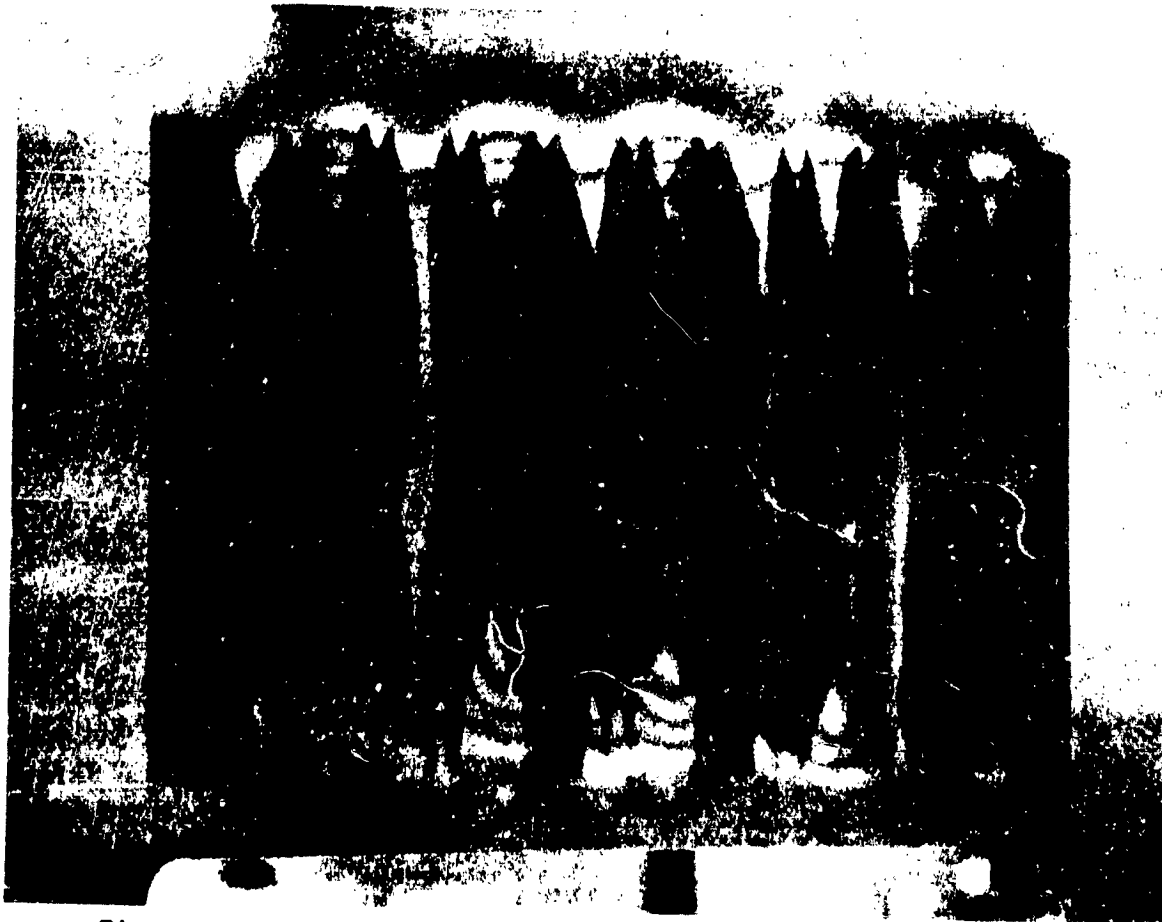


Figure 21. Outgassed Thin Film Pattern Behind Metallized Polyimide Films Irradiated for 1100 hours.

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is a reproduction of the patterns of outgassed molecules. Close examination of the original photos shows the patterns can be related to polyimide sample placement and subsequent twisting during the 1100-hour irradiation period. Even the elevated positions of the 1-gram weights below the shortest (Ciba Geigy) metalized samples can be discerned from a close examination of the photos.

The emittance of the thin film pattern of outgassed molecular matter was not measured. It has been estimated, however, as being substantially greater than the emittance of the nickel-plated, water-cooled sample block. The small  $4^{\circ}\text{C}$  difference in calorimeter equilibrium temperatures when the cases of samples present and samples absent are compared shows that the array of narrow, twisted test samples following 1100 hours of irradiation had net radiative exchange characteristics not unlike the outgassed thin film pattern.

From the test chamber standpoint, further control over the reflective and radiative properties of surfaces surrounding test specimens should be exercised during future experiments of the type performed for this program. Boeing, in fact, is already developing improved test techniques and configurations, including use of wider samples and chamber surfaces with controlled reflectance (Reference 3).



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3. Fogdall, L. B., and S. S. Cannaday, "Study of Front-Surface Aluminized Kapton Films Under Combined Electron, Proton, and Ultraviolet Radiation." Boeing Document D180-24073-1 in preparation.